

EA-6010-28
Dist. Category UC-90C

MATERIALS RESEARCH FOR THE CLEAN UTILIZATION OF COAL

Quarterly Progress Report

April - June 1981

S. J. Schneider
Project Manager

Center for Materials Science
National Bureau of Standards
U. S. Department of Commerce
Washington, D. C. 20234

PREPARED FOR THE UNITED STATES DEPARTMENT OF ENERGY

Office of Advanced Research and Technology

Under Contract No. EA-77-A-01-6010

"This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, expressed or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights."

TABLE OF CONTENTS

	PAGE
I. SUMMARY OF PROGRESS TO DATE.	1
II. DETAILED DESCRIPTION OF TECHNICAL PROGRESS	2
1. Materials Performance and Properties	2
2. Creep and Related Properties of Refractories	24

I. SUMMARY OF PROGRESS TO DATE

Brief Summary

1. Materials Performance and Properties

The major effort this quarter has been concentrated on the book "Construction Materials for Coal Conversion--Performance and Properties Data". The status of the various subsections of Section A (Materials Considerations and Performance Data) is: 1) "Operating requirements" - completed, 2) "Performance Data" and "Candidate Materials" - being drafted in final form. The assembling of test data for Section B is essentially complete and analysis of this data is in progress. An example of such an analysis is included in Appendix I. Background and status of the computerized data bases is given in Appendix II. The Data Center has transmitted 1100 computer abstracts of failure events and ten hard copy reports in response to queries during this quarter.

2. Creep and Related Properties of Refractories

Data was obtained on the creep of a fused cast $\alpha+\beta$ alumina (Monofrax A) under thermal cycling conditions and on silicon nitride using both linear variable differential transformers and specimen dimension measurements.

II. DETAILED DESCRIPTION OF TECHNICAL PROGRESS

1. Materials Performance and Properties (H. M. Ondik, B. W. Christ, A. Perloff, W. A. Willard)

Progress: During the last quarter the Data Center has received five requests for information and has responded by providing 1100 computer abstracts of failure events and analyses and ten hard copy reports.

The major thrust of the Data Center effort this last quarter has been the work on the book "Construction Materials for Coal Conversion--Performance and Properties Data" in order to deliver the draft to DoE by the end of the fiscal year. Completion of data tables for Section B, "Materials Testing Results", and assembling these pages with appropriate headings and Tables of Contents has been a massive job. Section B contains the summary tables and graphs of data abstracted from the DoE materials research contractors' reports. Just one section, that on corrosion in alloys, consists of over 120 tables of graphical summaries, many of which are contained on several pages, from 2 to almost 20 pages. Each of the subsections will have a separate Table of Contents in addition to the main Table of Contents at the beginning of the book. The main Table of Contents will contain only the major headings, the contents of each smaller section being too voluminous to permit inclusion and still have the Table convenient and usable.

Page headings for each page have been designed and are being entered on each page of the book. These headings will identify the section of the book to which that page belongs. For instance, the following are typical page headings chosen at random:

A.1 Coal Handling and Preparation Equipment	A.1.1
A.1.1 Conveying Equipment	page 1 of 1 - 6/81
=====	
A.2.2 Refractory Linings and Components--Dry-Bottom Vessels	A.2.2.2.1.4
A.2.2.2 Performance Data	page 1 of 1 - 6/81
=====	
B.1 Corrosion Effects, Chemical Reactions, and Phase Changes	B.1.1.15
B.1.1 Alloys	page 2 of 7 - 6/81
=====	
B.1 Corrosion Effects, Chemical Reactions, and Phase Changes	B.1.2.16
B.1.2 Refractories	page 2 of 10 - 6/81
=====	
B.2 Erosion, Erosion/Corrosion, and Abrasion Effects	B.2.1.21
B.2.1 Alloys	page 1 of 2 - 6/81
=====	
B.3. Mechanical Properties Testing	B.3.2.10
B.3.2 Refractories	page 1 of 1 - 6/81
=====	

The identification of pages by such page headings will enable the user who wishes to find specific tables, perhaps having first looked for a specific material in the index, to keep track of the location of sections and tables within the book.

Work on the index is progressing well. Since the index, which will enable users to find data for specific materials and specific properties, is so vital to the usefulness of such a book, great care must be taken in its preparation. The subsections of Section A, "Materials Considerations and Performance Data", are in varying stages of preparation. The "Operating Requirements" subsections are complete, while the "Performance Data" and "Candidate Materials" subsections are being drafted in final form. The analysis of data from Section B is progressing. An example of such an analysis, taken from the area of mechanical properties, is in Appendix 1.

Because of the enormous amount of effort needed to assemble the book in final form, work on the computer data bases has lagged somewhat. Keyboarding of masses of data has not begun, only small portions of test batches having been entered. In order to save computer storage costs, little of the Materials Properties Data Base is being retained in the computer at this time. The Materials and Components Plant Performance Data Base is, of course, being maintained fully in order to answer queries and enter new information as it is submitted.

Appendix 2 constitutes a report on the background and current status of the Data Base design for both data bases, Properties and Plant Performance, as required by our work statement.

Plans: During the next quarter, the final writing of sections and assembling of the book "Construction Materials for Coal Gasification--Performance and Properties Data" should be completed. The usual routine work of cataloging reports, abstracting data, and answering queries will continue.

Appendix 1

Example of a section A "Materials Evaluation" Segment. The accompanying B section pages are not included.

Mechanical Properties of Metal Alloys Used

In Coal Gasification System

Mechanical properties, such as tensile test results, hardness, and fatigue crack growth rates for metal alloys used in coal gasification systems are reviewed in this section. The focus is on nickel alloys, 2 1/4 chrome-1 molybdenum steels, stainless steels and low-carbon steels in various environments, including hydrogen sulfide, hydrogen, dry argon, vacuum and typical coal gasification atmospheres. Data tabulations appear in Sections B.3.1.8 through B.3.1.12 and B.4.1.1 through B.4.1.3.

Some uniaxial tensile data on four alloys tested at elevated temperatures appear in Table B.3.1.8. The yield and tensile strengths of each alloy decreased with increasing temperature. The elongation shows a mixed response to increasing temperature. In some cases it increased and in others it decreased.

Some high temperature tests were performed to evaluate the effect of exposure to typical coal gasification atmospheres on the uniaxial tensile properties of four alloys (Table B.3.1.9). Exposure was for 100 hours at pressures of 500 and 1500 psi (34 and 102 atmospheres). Temperatures were 1382, 1600 and 1800 °F. Two typical coal gasification atmospheres were used. The gas compositions for each combination of pressure and temperature appear in Table B.3.1.9. Hydrogen sulfide concentration ranged between 1 and 2 percent and was close to 1% for atmosphere number 2. Following exposure, specimens were removed from the atmosphere and pulled to fracture under dry-flowing argon in a constant crosshead speed testing machine at a temperature of 1382, 1600 or 1800 °F. Specimen design conformed to ASTM Designation E8. The following tensile properties were measured: yield strength, tensile strength, uniform strain and total elongation in a two inch gauge length. Test results appear in figures 1 through 4, along with a reference value determined at temperature on an unexposed specimen.

In general, these results show that a 100-hour exposure to the coal gasification atmospheres does not significantly affect the stability of the tensile properties of the four alloys tested. Property changes are small, but nevertheless, noticeable. Properties showed both increases and decreases. In general, elongation changed more than strength. For example, the maximum change in total elongation was a 50% decrease, whereas the maximum change in yield strength was a 30% increase. Most of the observed changes in tensile properties were smaller, e.g., $\pm 10\%$. Figures 1 and 2 show that the yield and tensile strengths of each alloy decreased with increasing temperature. The elongation shows a mixed response to increasing temperature for all four alloys. In some cases it increased and in others it decreased. These results are the same as for unexposed specimens tested in vacuum (see Table B.3.1.8), indicating that exposure to typical coal gasification atmospheres for 100 hours does not have a major effect on the temperature dependence of tensile properties for these alloys.

Figures 1 and 2 show that effects of the pressure of the coal gasification atmospheres, 500 vs. 1500 psi, on the observed strength changes, were small. Strength-change differences between the 500 and 1500 psi tests were about $\pm 10\%$. Elongation-change differences between the 500 and 1500 psi tests were somewhat larger, i.e., about $\pm 20\%$ (Figure 4).

Some 1000-hour aging treatments in vacuum were carried out at 1382, 1600 and 1800 °F, on some untreated test specimens to facilitate a comparison of tensile property changes due to aging and to typical coal gasification atmospheres (Table B.3.10). Exposure times were 1000 hours in vacuum and in the coal gasification atmospheres. Two coal gasification atmospheres were used at pressures of 500 and 1500 psi. The gas compositions for each combination of pressure and temperature appear in Table B.3.10. Hydrogen sulfide ranged between 1 and 2 percent, and was closer to 1% for atmosphere number 2. Specimen design conformed to ASTM Designation E8. Specimens were pulled to fracture under dry flowing argon in a constant crosshead speed testing machine. Properties measured were ultimate tensile strength, uniform strain and total elongation.

Test results appear in figure 5. The ratio of the property of the exposed specimen to the property of the aged specimen has been plotted. Values greater than one indicate an increase in the property of the exposed specimen relative to the aged specimen, and vice-versa. In general, the ratio for uniform strain showed a greater change with exposure than did the ratios for total elongation and tensile strength. Inconel 671 and USS 18-18-2 showed the largest increases in the ratio for uniform strain, with the largest ratio being 4.9. About half the tests for uniform strain showed a decrease in the ratio, with the lowest ratio being 0.4.

Effect of Temperature and Environment on Fatigue of 2 1/4 Cr-1 Mo Steel

Fatigue crack growth kinetics of 2 1/4 Cr-1 Mo steel were evaluated in various environments and over a range of temperatures and pressures. The test environments were: vacuum, dehumidified argon, water vapor, dehumidified hydrogen and hydrogen sulfide. Temperatures ranged between 295 and 477 °K. Pressure varied between 0.1 and 5.0 torr in some tests. Results appear in B.3.1.11 and B.3.1.12. These figures show that crack growth rate was in the range 10^{-7} to 10^{-5} inches per cycle for $20 < \Delta K < 70$. Increasing frequency from 5 to 10Hz increased crack growth rate slightly. Crack growth rate increased with increase in stress intensity factor range. For a fixed value of stress intensity factor range, crack growth rate generally decreased with increase in temperature. Hydrogen sulfide atmospheres caused fastest crack growth rates, and rates increased with increasing hydrogen sulfide pressure. Dehumidified hydrogen caused the second fastest crack growth rates. Water vapor, dehumidified argon and vacuum tests resulted in comparable crack growth rates at $\Delta K > 50 \text{ MPa-m}^{1/2}$.

Effects of Exposure to Flue Gases from Four Different Coals

Six alloys, one clad alloy and 5 combinations of welded alloys were exposed to flue gases from four different coals to determine how the different gases affected the materials. Depth of corrosive attack and/or hardness were measured to evaluate changes in the alloys. The test specimens were 2-inch O.D. pipe sections threaded together to form two lengths of pipe, one of the

welded alloys and one of the remaining alloys. The pipe exteriors were exposed to the flue gases in coal-fired commercial steam generators. Air flowed inside the pipes to control temperature. Temperatures ranged from 800 to 1440 °F. Exposure times were from 3144 to 7368 hours. Data appear in Tables B.4.1.1 and B.4.1.3. Tables I and II show some of the trends detected for the influence of temperature and exposure time on severity of corrosive attack or hardness changes. Severity has been rather arbitrarily classified as low, medium or high, using the full range of depth of attack in Table B.4.1.1 as the basis. Table B.4.1.3 shows that weld hardness for three of the five combinations of welded alloys investigated is significantly higher than for the other two. This table also shows that lack of penetration is common to all five combinations of welded alloys.

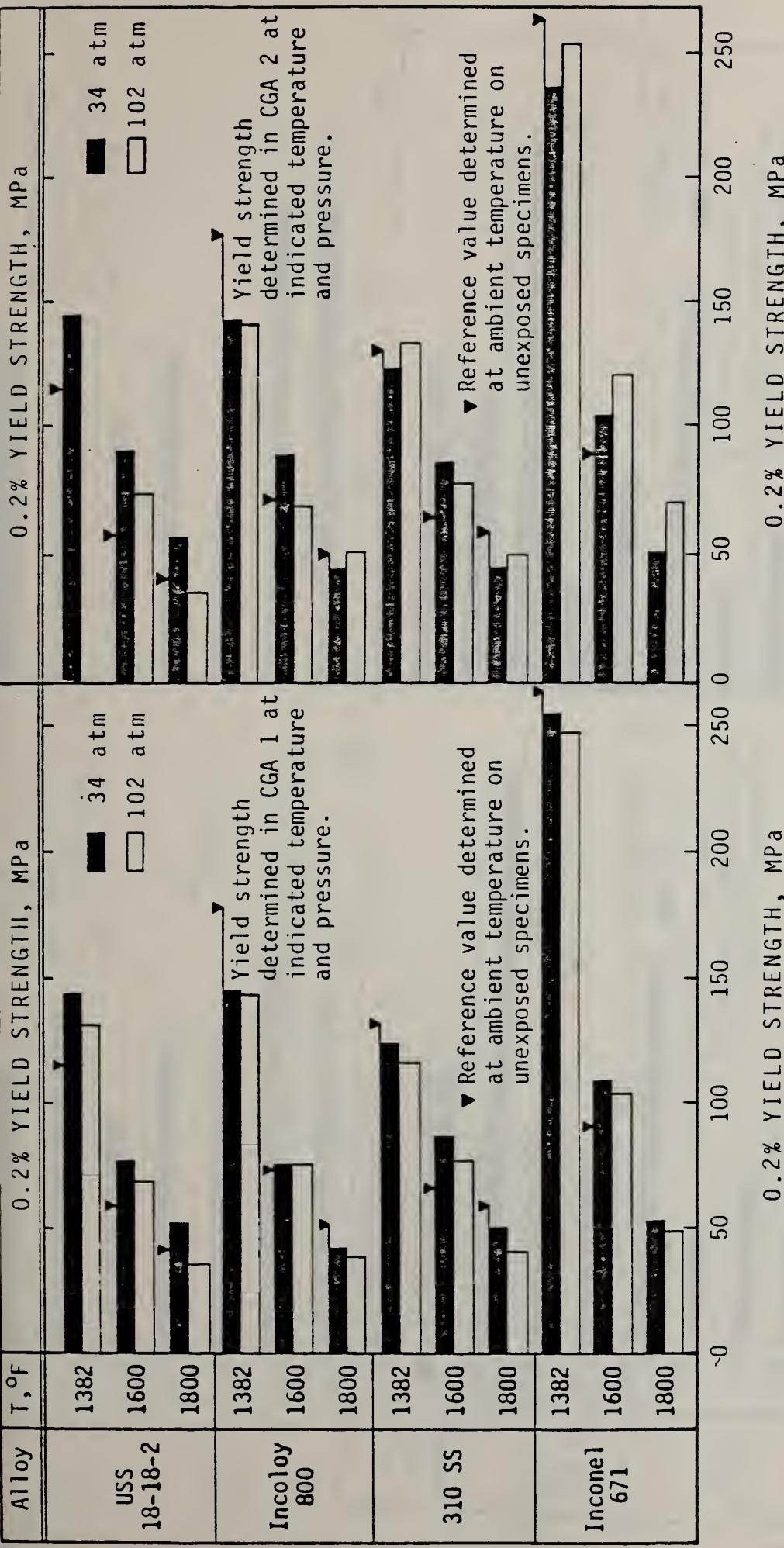


FIGURE 1

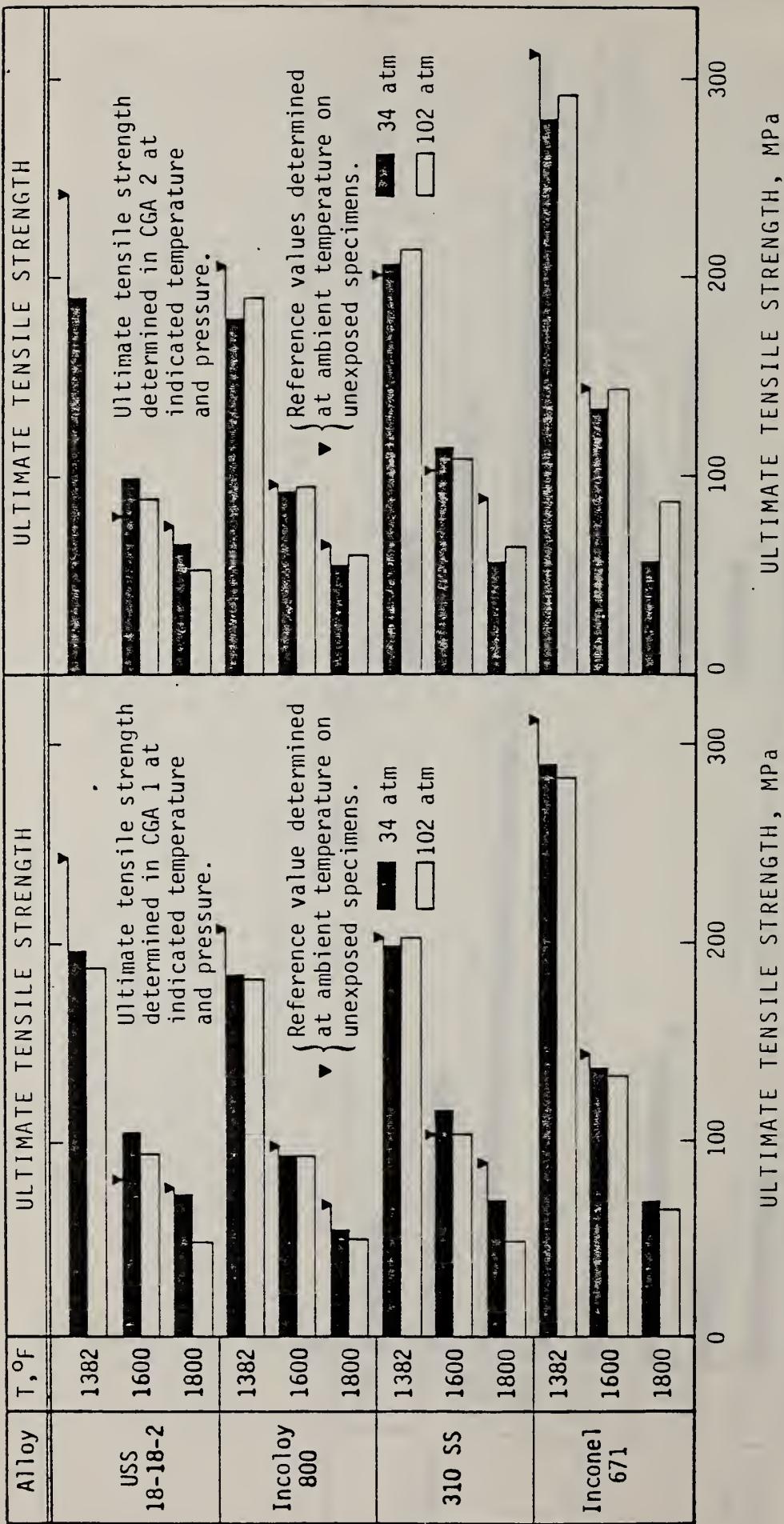


FIGURE 2

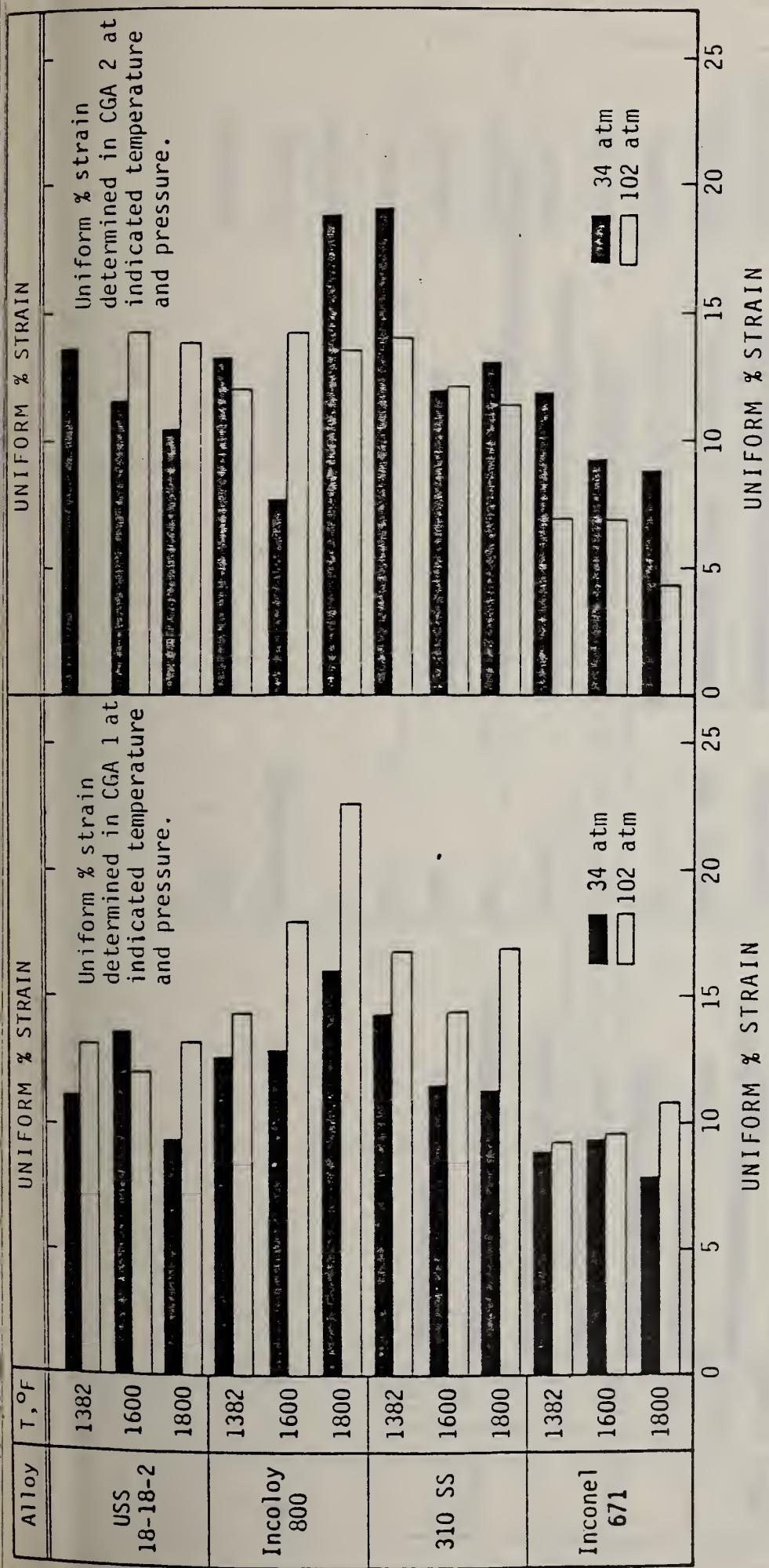


FIGURE 3

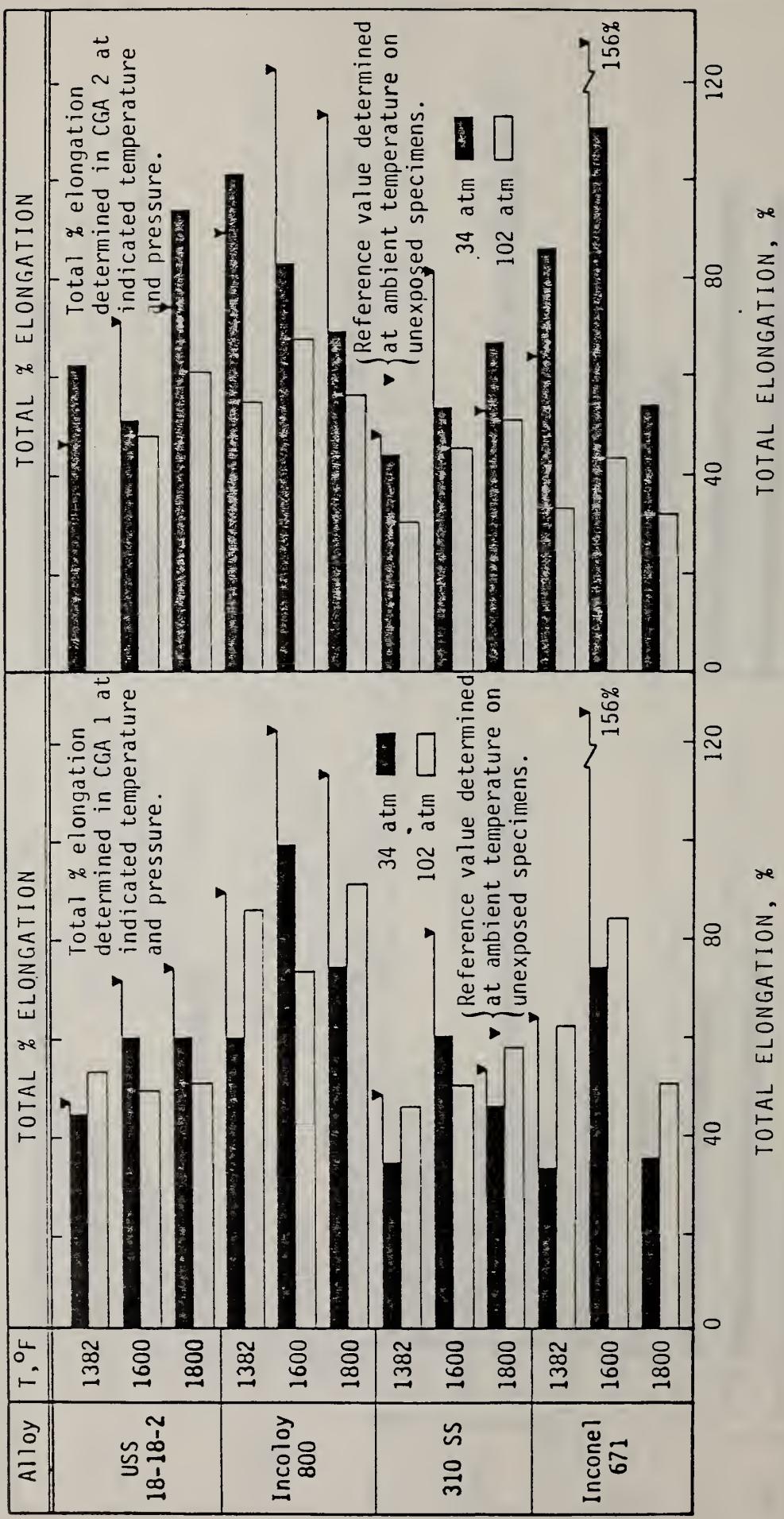


FIGURE 4

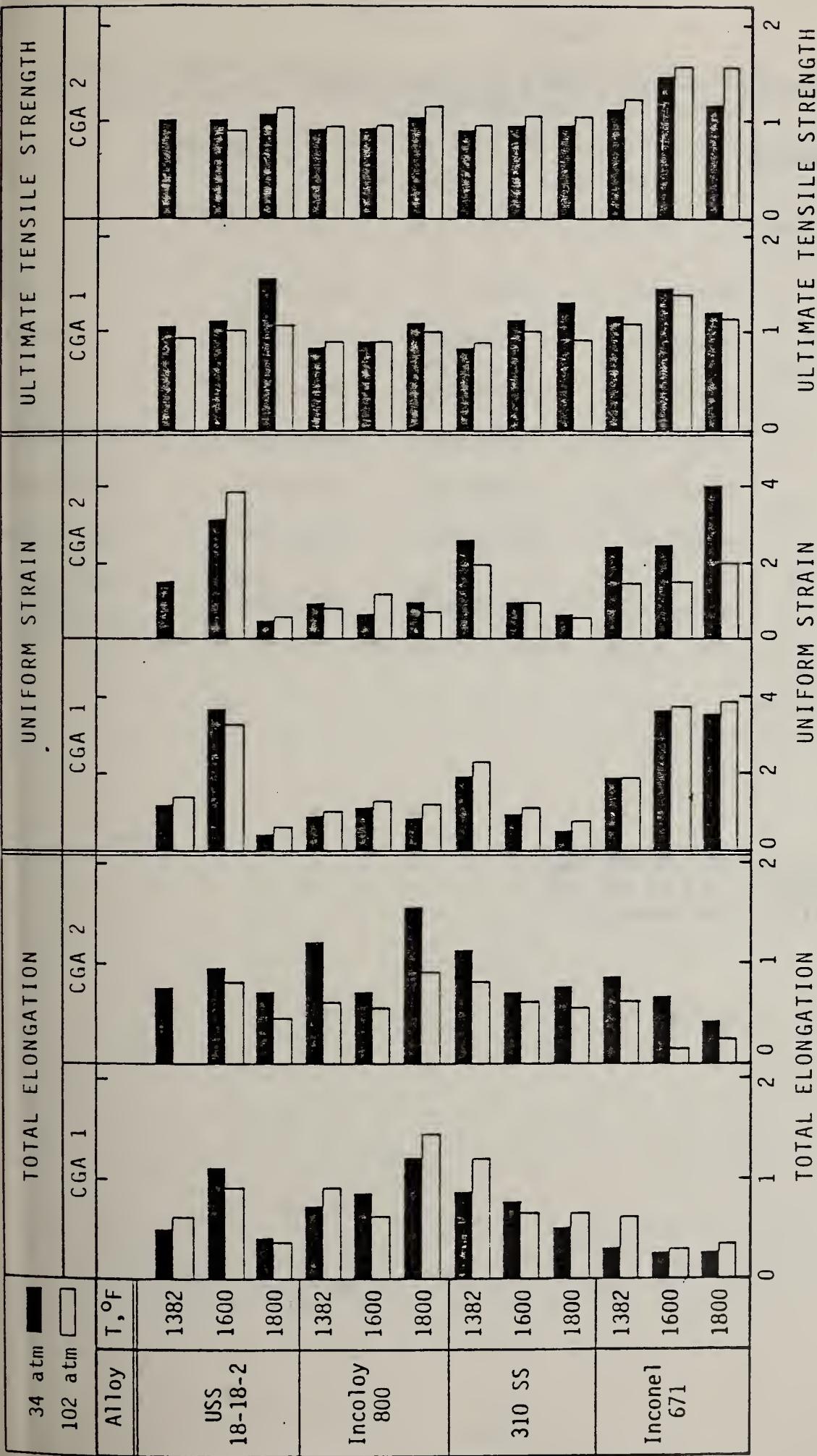


FIGURE 5

TABLE I
Severity of Corrosive Attack of Several Alloys
by Flue Gases From Four Coals

	Subbitum. C	HV Bitum. C	HV Bitum. A	Lignite A
316SS	L-M*	M	L-M	L
310SS	L	L	L	L
12R72	L	L	L-M	M-H
Incoloy 800	L-M	L-M	M-H	L
Inconel 617	L-H	L-M	L	L
Haynes 188	L-H	L-M	L	L
Inconel 671	H	L-M	L	L-H
Clad on In 800				

*L - Low severity - 0 to 2.5 mils deep.
 M - Medium severity - 2.5 to 4.5 mils.
 H - High severity 4.5 to excessive.

Table II

Trends in hardness changes for alloys exposed to flue gases in coal-fired commercial steam generators.

Hardness Change After Flue Gas Exposure From Each Type of Coal

ALLOY

	Subbituminous C	HV Bituminous C	HV Bituminous A	Lignite A
316 SS	increased	decreased	increased	increased
310 SS	decreased	decreased	increased	decreased
12 R 72	decreased	decreased	increased	decreased
Incoloy 300	increased	decreased	increased	decreased
Inconel 617	decreased	increased	increased	increased
Haynes 188	increased	increased	increased	increased
Inconel 671 clad on Incoloy 8u0	decreased	decreased	no change	decreased

Appendix 2

Development and Design of Compatible Data Bases for Materials and Components Plant Performance and for Materials Properties

The Materials and Components Plant Performance Data Base has operated since 1976 (under the title of Failure Information). The program began with the submission to DoE and NBS of reports of operating events at coal gasification and liquefaction pilot plants and development units. These operating event reports, usually of failures of materials and components, were carefully reviewed and the data abstracted and summarized by NBS staff experts in materials. Frequently, the reports of the event are followed by comprehensive diagnostic failure analyses which often contain recommendations for future failure avoidance, and these reports are also abstracted and summarized. These abstracts, containing all the pertinent information in the reports, were maintained as a data base in computer storage using a Data Base Management System (DBMS) available on a government agency computer which was satisfactory for the simple structure of the data base. The data stored in the computer consists of the abstract as a block summary along with some nine keywords which identify the abstract and permit the data to be retrieved by the use of a variety of the keywords. The data base can be searched and the file sorted by one or any combination of these keywords--material name, component name, process or plant name, failure mode, etc. The structure of this data base is entirely suitable and no revision has been considered necessary.

The requirements of the Materials Properties Data Base, however, could not be supported by the simpler DBMS being used by the Plant Performance Data Base. The design of The Materials Properties Data Base has evolved over a period of time although the complex structural relationships between data items demanded by the nature of the data have not changed. The data are so varied in kind and style that a very flexible computer system is required to handle them.

The numeric data include single-value numbers, ranges of numbers, and values taken by digitizing values from graphs and histograms. Descriptive textural information must also be accommodated in the computer. Along with the data the specific experimental conditions must be stored as well as pertinent facts about the test procedure used. Each material must be designated not only by appropriate synonyms but also by the processing methods used, including its thermomechanical history. The data base must be of such a design that it is open-ended with respect to the addition of new types of data as well as possible multiple entries from different sources for similar information. Full bibliographic reference information must also be included.

Data Center staff personnel have had the benefit of Data Base Management System (DBMS) courses as well as practical demonstrations of a number of DBMS by computer vendors. The first class was a special three-day general course taught by Professor E. Sibley of the University of Maryland. Staff were introduced to the characteristics of DBMS, the great variety, advantages and disadvantages of the various types of DBMS, and were also given a good many caveats concerning the choice and use of such systems. We also had the benefit of information in an NBS publication which compared several DBMS

(NBS Technical Note 887, "Six Data Base Management Systems: Feature Analysis and User Experience", by E. Fong et al., November 1975), as well as a number of NBS Special Publications dealing with general information about DBMS and interactive computer services. Using the knowledge thus gained the staff decided to put further study into a specific major DBMS which has a hierarchical structure. A week's course was taken and a data base was designed establishing the data relationships required. A full description of this data base design was reported in report EA-6010-18, "Materials Research for the Clean Utilization of Coal", Quarterly Progress Report, October-December 1978.

A sample data base was set up using the hierarchical DBMS under a temporary contractual arrangement. Testing of this sample materials properties data base revealed that, although data storage was relatively economical in the hierarchical structure which eliminated the necessity to repeat any information, the query system was very complicated. Questions had to be very carefully, often intricately, worded to insure the correct searches and answers. This system would prove expensive in personnel time for use in our work, and we considered it unsuitable for further serious consideration.

The sample data base and structure of this hierarchical system was extremely valuable in providing a basis from which the competing vendors could work in setting up demonstrations of their ability to cope with our data and in performing benchmark tests upon which costs were based in awarding the contract. Data Center Staff did have to expand the amount and kinds of data in the base to provide for benchmark tests of realistic complexity. The procurement procedure was a prolonged one, partly because of the fact that commercial vendors are well-acquainted with business-oriented data bases but have little or no experience with the kind of complex technical data with which we deal.

Services of a commercial computer vendor were procured which enable us to fulfill the complex computer requirements as well as the need to comply with the sponsor's request that we provide for future direct on-line access capability for non-NBS users. The services include direct computer access through a nationwide network and suitable computer software for managing the data base in the form of a Data Base Management System. The DBMS now in use is well-known as a "user-friendly" system, a real advantage for future direct searching access by non-NBS users.

As reported in the January-March 1981 quarterly report, the Materials and Components Plant Performance Data Base, having already been established within a different DBMS, was transferred to the vendor's computer on the new DBMS, maintaining the same capabilities for search and the same structural relationships. The Plant Performance Data Base is operating on this new system. The Materials Properties Data Base is being designed to be compatible with the Materials and Components Plant Performance Data Base and will be keyboarded as rapidly as possible.

Data bases are characterized in structure as hierarchical, relational, or network. The DBMS we have contracted to use is known as associative in that it can support any one or any combination of the three characteristics. It is unique among DBMS in that several data bases can be searched in one query, the

separate nature of the several bases being "invisible" to the user. This last characteristic is especially of interest for us because of the usefulness of searching the Materials and Components Plant Experience Data Base and the Materials Properties Data Base simultaneously. The two bases will be searchable through these common elements:

- material names (both data bases)
- component names (both data bases)
- failure mode (plant performance base) and testing behavior (properties base) -- an example here are terms such as corrosion or erosion.

There are two major search patterns which may be followed to retrieve items in the Data Bases under the control of the DBMS language. It is possible to search a given data item for a certain sequence of characters which may be the entire content of the data item or only part. Such sequential searching is not the fastest way to find data although demonstrations have shown it to be practical for small amounts of information. It is more efficient to make certain parts of the data bases directly accessible by setting up indexes of certain records, that is, by designating data items as "keyed". Search for the data items is therefore more direct, and more rapid.

The Materials and Components Plant Performance Data Base structure, essentially unchanged even though the data are now under the control of the new DBMS, is defined in the new DBMS by the following table, known as a Fields Definition Table (FDT). The transfer from one DBMS to the other was performed by the computer vendor.

Materials and Components Plant Performance Data Base FDT

<u>Field Name</u>	<u>Key</u>	<u>Type</u>	<u>Stored Length</u>	<u>Structure</u>	<u>Repeats</u>	<u>Notes</u>
INFONO		PFX	CHR	5	BASE	SCALAR
INFONUM			UNP	5	SUBF	SCALAR
INCIDENT		PFX	CHR	5	BASE	SCALAR
INCIDNUM			UNP	5	SUBF	SCALAR
DATE		PFX	CHR	6	BASE	SCALAR
DATENUM			UNP	6	SUBF	SCALAR
PROCESS		PFX	CHR	15		SCALAR
LAB		PFX	CHR	20		SCALAR
REPORT		PFX	CHR	10		2
AUTHOR		PFX	CHR	V 30		5
MATERIAL		PFX	CHR	V 30		10
FAIL1		PFX	CHR	V 30		SCALAR
FAIL2		PFX	CHR	V 30		5
COMP1		PFX	CHR	V 30		SCALAR
COMP2		PFX	CHR	V 30		4
TEMP			UNP	4		SCALAR
PRESSURE			UNP	4		SCALAR
ATM		PFX	CHR	30		3
SUBSYS		PFX	CHR	30		3
REFER			CHR	V 305		SCALAR
SUMMARY			CHR	V 5000		SCALAR

Explanation of terms in the above Table:

INFONO = the NBS file number which corresponds to a number assigned to the original report received

INFONUM = an identifier which makes the INFONO field searchable as a numeric field

INCIDENT = a number assigned to a given plant event within a report (a report may provide information about several incidents)

INCIDNUM = an identifier which makes the INCIDENT field searchable as a numeric field

DATE = date entered on the report

DATENUM = an identifier which makes the DATE field searchable as a numeric field

PROCESS = name of the plant and/or the process

LAB = origin of the report

REPORT = type of report (example: failure event, failure analysis, materials evaluation)

AUTHOR = name(s) of writer of the report

MATERIAL = name(s) of the material(s) in the failed component

FAIL1 = the general cause of failure (example: corrosion)

FAIL2 = the specific cause of failure (example: sulfidation)

COMP1 = general terminology regarding the component (example: pumps, valves)

COMP2 = specific failed part (example: bearings housing, gate)

TEMP = temperature at which the component operated

PRESSURE = pressure at which the component operated

ATM = the environment surrounding the component (example: composition of gaseous atmosphere)

SUBSYS = the area of the plant in which the component part is situated (example: gasifier vessel, coal handling)

REFER = the full reference for the report, title, author(s), date, etc.

SUMMARY = abstracted summary version of the report

Data Center staff have participated in two courses, one to familiarize us with the overall computer control system used by the vendor and one which trained us in the use of the DBMS. It was this latter course, combined with consultation with the vendor's systems analysts, which permitted Data Center staff to arrive at the current design for the Materials Properties Data Base.

The current design of the Materials Properties Data Base is dependent on the multiple data base capability of the DBMS. The Materials Properties Data Base will actually consist of three data bases, which can be searched separately or as one base. The ability to cross-link the information in the multiple data bases permits the setting up of information in such a way that data items need not be repeated at all or only a minimum number of times. Computer storage charges are greatly lowered thereby. It is possible to allow for a variable length field for a data item (such as comments) up to a very large maximum. A variable number of repeat fields may also be allowed for a data item (such as synonyms), again up to a large maximum. Since the DBMS structure allows for this assignment of space to data items without payment of storage charges for unused space there is relative freedom and good flexibility in planning for data items in the data base. The three data bases of the present design also carry data items which are identifiers linking the three, simple 8 character words which provide the search links. These identifiers are keyed items. Small amounts of data have been tested. The keyboarding procedures have been programmed for our computer terminals and entering data will be relatively simple. This DBMS is "user friendly" at the input end of the data effort as well as the search and output end.

References will be stored in one of the data bases. The data items to be included in this base are: report or article title, author's names, full reference, report number, contractor name, contractor address, contract number, keywords. A definition of the structure of the reference Data Base is contained in the following Fields Definition Table.

REFERENCES DATA BASE FDT

<u>Field Name</u>	<u>Key</u>	<u>Type</u>	<u>Stored Length</u>	<u>Structure</u>	<u>Repeats</u>	<u>Notes</u>
REPTID	PFX	CHR	8			scalar
CONTRCTR	PFX	CHR	8			scalar
CONTRNAM		DCD	V 150			scalar)
CONTRLOC		DCD	V 150			decode on CONTRCTR
TITLE		CHR	V 200			scalar
AUTHORS		CHR	V 200			scalar
CONTRNUM		CHR	V 30			scalar
REPTNUM		CHR	V 20			scalar
REFERENC		CHR	V 200			scalar
NTISNUM		CHR	V 30			scalar
KEYWORD		CHR	20		V 15	

Explanation of terms in the above Table:

REPTID = an internal identification number for the specific reference

CONTRCTR = an abbreviated description for the contractor

CONTRNAM and CONTRLOC = full name and address of the contractor (these items are in a separate file called a Decode file. They can be called up by the CONTRCTR entry. CONTRCTR is a maximum of 8 characters and is repeated with each reference, but the full name and address need be entered and stored only once no matter how many reports belong to the same contractor)

TITLE = title of the report

AUTHORS = all authors named for the report are included

CONTRNUM = the number of the contract supporting the work of the report

REPTNUM = the number of the individual report

REFERENC = the kind of report (i.e., annual, quarterly, etc.), the date and year, the name of the sponsor, etc.; full identification of the report

NTISNUM = the number by which the report can be obtained from NTIS

KEYWORD = up to 15 different words or phrases can be accommodated in this field and can be searched

The PFX in the Key column indicates that that is a "keyed" item. The CHR in the Type column indicates a character field (as opposed to numeric); DCD in the same field indicates DECODE field items as explained above. A V in the Stored Length field indicates that the field length is not fixed but variable and may contain any number of characters up to the stated maximum. Under the Repeats column, the word scalar indicates that there is no repeat of that data item; V followed by a number indicates that a variable number of such data items may appear up to the stated maximum.

Materials designations will form the second base: material type, material name, synonyms, composition in terms of constituents and the amounts, preparation, characteristics, manufacturer, and a comments item for any necessary clarification of any of the above. The Fields Definition Table for this data base follows.

Materials Data Base FDT

<u>Field Name</u>	<u>Key</u>	<u>Type</u>	<u>Stored Length</u>	<u>Structure</u>	<u>Repeats</u>	<u>Notes</u>
MATLID		PFX	CHR	8		scalar
REPTID		PFX	CHR	8	V 500	
PROPID		PFX	CHR	8	V 500	
MATLTYP		PFX	CHR	V 70	scalar	
MATLNAME		PFX	CHR	V 35	scalar	
NAMELIST		PFX	CHR	35	V 10	
COMPSTN			CHR	32	Base	V 50
CONSTIT		PFX	CHR	20	Subf	V 50 (1-20)
AMOUNT			CHR	12	Subf	V 50 (21-32)
AMTNUM			UNP	6	Subf	V 50 (21-26)
AMTUNIT			CHR	6	Subf	V 50 (27-32)
MATLPREP			CHR	V 1000	scalar	
MATLCHAR			CHR	V 1000	scalar	
MANUFAC			CHR	V 70	scalar	
COMMENTM			CHR	V 1000	scalar	

Explanation of terms in the above Table:

MATLID = an internal identification number for the material

REPTID = an internal identification number for the specific reference

PROPID = an internal identification number for the property or performance item

(Note: MATLID, PROPID and REPTID are the items through which the data bases are linked)

MATLTYP = material type, i.e., alloy, refractory, etc.

MATLNAME = name of the material

NAMELIST = MATLNAME plus synonyms

COMPSTN = composition of the material. This field is a base field which consists of the four subfields which follow and which are all individually searchable. The Notes column indicates which characters within the COMPSTN field form the individual subfield

CONSTIT = the chemical constituents of the material

AMOUNT = the amount of the constituent in the material

AMTNUM = an identifier which makes the AMOUNT field searchable as a numeric field

AMTUNIT = designation of the units belonging to amount, i.e., weight percent, mole percent, etc.

MATLPPREP = the method of preparation and treatment of the material, thermomechanical history, etc.

MATLCHAR = the characterization of the material

MANUFAC = manufacturer of the material

COMMENTM = any comments concerning any of the above items about the material

The actual properties or test results will be in a third base: material name, component name, name and units of the property, test conditions, test results or value(s) of the property; this last actually consists of a large number of data items to accommodate the complex and varied nature of the data we must include: a comments item will also be included in this base to allow space for any necessary clarification. The current design of the properties data base is outlined in the following table.

Properties Data Base FDT

<u>Field Name</u>	<u>Key</u>	<u>Type</u>	<u>Stored Length</u>	<u>Structure</u>	<u>Repeats</u>	<u>Notes</u>
REPTID		PFX	CHR		8	V 500
MATLID		PFX	CHR		8	V 500
PROPID		PFX	CHR		8	scalar
MATLNAME		PFX	CHR	V	50	scalar
COMPNAME		PFX	CHR		30	V 10
PROPNAME		PFX	CHR	V	50	scalar
PROPUNIT			CHR	V	50	scalar
TESTCOND			CHR	V	1000	scalar
COMMENTP			CHR	V	1000	scalar
RSLTTEXT			CHR	V	1000	scalar
RSLTEQUA			CHR	V	500	scalar
VARIABLE			CHR		30	V 6
VARNAME		PFX	CHR		20	Subf V 6
VARUNIT			CHR		10	Subf V 6 (1 to 20)
VARDATA			CHR		60	Base V 200
VAR1NUM			UNP		10	Subf V 200 (21 to 30)
VAR2NUM			UNP		10	Subf V 200 (1 to 10)
VAR3NUM			UNP		10	Subf V 200 (11 to 20)
VAR4NUM			UNP		10	Subf V 200 (21 to 30)
VAR5NUM			UNP		10	Subf V 200 (31 to 40)
VAR6NUM			UNP		10	Subf V 200 (41 to 50)
VAR1ALFA			CHR		10	Subf V 200 (51 to 60)
VAR2ALFA			CHR		10	Subf V 200 (1 to 10)
VAR3ALFA			CHR		10	Subf V 200 (11 to 20)
VAR4ALFA			CHR		10	Subf V 200 (21 to 30)
VAR5ALFA			CHR		10	Subf V 200 (31 to 40)
VAR6ALFA			CHR		10	Subf V 200 (41 to 50)
						(51 to 60)

Explanation of terms in the above Table:

REPTID, MATLID, PROPID and MATLNAME have been defined above

COMPNAME = name of the plant component or plant use area for which the material property was studied

PROPNAM = the name of the property or performance item

PROPUNIT = the units in which the data are reported (Data Base is to be standardized on SI units)

TESTCOND = the experimental methods and conditions of the test

COMMENTP = any necessary comments about any of the items in this properties file

RSLTTEXT = any test results which is given verbally, i.e., non-numeric, qualitative, or descriptive

RSLTEQUA = any test result given in the form of an equation

VARIABLE = a repeating field which lists each test variable (such as temperature, pressure, gas composition, etc.) and their units (maximum of six variables)

VARNAME = subfield of "variable" containing the name of the variable, e.g., temperature

VARUNIT = subfield of "variable" containing units for the variable, e.g., kelvin

VARDATA = numerical data point consisting of up to six values, one for each parameter listed under variable

VAR1NUM = numerical value of first variable

VAR6NUM = numerical value of sixth variable

VAR1ALFA = alternative field in case first variable is non-numeric value (e.g., if first variable is "gas exposure", value might be "after")

VAR6ALFA = alternative field in case sixth variable is non-numeric value

There is a possibility that the data base may be altered somewhat to provide a fourth, simple base containing only test conditions. The advantage is the diminished amount of keyboarding and the smaller use of storage space since this item can be quite lengthy and must be repeated under the present structure. This alteration is simple at this stage and does not disturb the overall design of the Materials Properties Data Base.

Although the design of the Data Bases has so far accommodated the small amounts of test data, only a large scale test can confirm their suitability and permit permanent stabilization of the designs. It is anticipated that the Reference and Materials Data Bases will not change but that the Properties Data Base, being the most complicated portion, will have some changes and perhaps additional data items to be included.

Once the designs have stabilized the Data Center staff can focus on the instructions to be included in a user's Manual for non-NBS users. The queries required by the DBMS are very close to normal English and the rules are not difficult to learn. The user language of the DBMS permits selective retrieval of data and varied designing of report formats. Although some searches may be performed by using more than one manner of querying to obtain the same result, the staff will probably standardize only one for the outside user to simplify the situation. It is possible to design queries which are sequences of several command statements and to designate these sequences to be stored for later use. These commands are retrievable by an assigned label. The designated sequence is called a "macro" and the label is called a "macro name". These "macros" can be used over and over again when the same queries are desired to be used. Data Center staff will be able to design macros for non-NBS users use which will enter the query and produce a tabulated print out with very little trouble to the user. The user will not be required to learn about the detailed structure of the Data Bases or the various methods of searching or how to obtain the various styles of tabulated presentations of the results. These macros can be written so as to present the multiple data bases of our system as one base to the user. Although Data Center staff recognize that there must be a great deal of careful testing of the system and the instructions to be given in a User's Manual it is not anticipated that any non-NBS users will experience any great difficulty with a well-tested system.

2. Creep and Related Properties of Refractories (N. J. Tighe, C. L. McDaniel, S. M. Wiederhorn)

Progress: In order to evaluate the creep test facility capability for measuring specimen deflection continuously over a 1000 hour test period, specimens of silicon nitride from a well-characterized billet were tested in static loading in four-point bend configuration. Three linear variable differential transformers (LVDT) were mounted on the upper frame of one furnace in a position to contact the upper loading ram. In this position, we are measuring the difference between the specimen movement and the frame. This choice was made over an LVDT system in direct contact with a specimen because of the difficult contact problems and the cost of such a system. Since the objective of the present program is to carry out screening tests rather than precision creep experiments, the simple frame to push rod system appeared justified. We therefore used specimens for the initial tests for which we had some creep data for comparison.

The tests were run for 1000 hours at 1200 °C using 6 specimens 3 x 4 x 50 mm, three specimens were in the furnace with the LVDTs and three were in another furnace. The LVDTs were monitored continuously with multiple point recorders. We used the long test period to evaluate several recorders and, based on their performance, purchased a 12 point sequential recorder that was most satisfactory. This recorder should be delivered in late July. On the basis of the performance of the 3 LVDTs, we completed the purchase of the required 12 for the system and they will be ready in late July.

The outer fiber strain curve drawn from the deflections measured for the silicon nitride specimens is shown in Fig. 1. The data points were taken from the continuous recording at 1 hour intervals for the first 24 hours and at 24 hour intervals for the next 41 days. The deflections were similar for all of the specimens and the plots showed three regions of different deflection rate or creep as indicated on the diagram. The rates calculated for the two regions of constant creep are: Region II 6.9×10^{-8} /second, and Region III 2.3×10^{-6} /second.

Additional thermal cycling tests were carried out on the fused cast $\alpha+\beta$ alumina (Monofrax A) reduced, cross-section specimens tested in compression. We had planned to run for 1000 hours with cycling every 1/2 hour between 1400 °C and 1200 °C. However, the tests were terminated after one week because of a water-clog problem. During these tests we recorded the LVDTs and found that they cycled over ~.05 cm because of thermal expansion changes in the load train. A correction for the LVDT readings must be considered in the analysis of thermal cycling in our system. The cycles were symmetrical throughout the test interval and the end points could be averaged for a total deflection reading. In preparation for carrying out four-point bend tests on the alumina-type refractories we have had new fixtures made from the same recrystallized alumina material used for rams.

Plans: During the next quarter we will carry out static fatigue tests on the silicon carbide discussed in the previous report. Tests will be run for 1000 hours at 1200 °C and 1300 °C. Specimens of Monofrax A will be cut and tested in the four-point bend configuration in order to compare the material properties in this mixed mode with the simple compression results.

Because of the recent change in emphasis from refractories for MHD air preheaters to refractories for more general coal derived fuel applications such as heat exchangers and vessel liners for gasifiers and liquefaction systems, we are including some of these materials in our screening program.

The following materials are of interest for structural refractories in fossil energy generating systems for insulating liners, structural supports, and heat exchanger tubing. For some of the materials there is data on coal slag corrosion resistance but there is very little data on the strength or creep resistance of the materials under the types of stresses they would be exposed to for periods of thousands of hours. Some ranking of the materials with respect to creep resistance in air at temperatures up to 1600 °C for periods of up to 1000 hr. will be required for future design purposes. We plan to include some of these materials in our testing program.

Oxide ceramics, fused cast:

α -alumina: $\alpha+\beta\text{Al}_2\text{O}_3$, 92-95% $\alpha\text{Al}_2\text{O}_3$ and 5-8% $\beta(\text{Na}_2\text{O}\cdot\text{Al}_2\text{O}_3)$ (Monofrax A)

β -alumina, Al_2O_3 ($\text{Na}_2\text{O}\cdot\text{Al}_2\text{O}_3$)

Magnesia-spinel, $(\text{MgO}\cdot\text{Al}_2\text{O}_3)$ + MgO

Chrome-spinel, 80% $(\text{MgCr})\text{O}_3$ + 15% (Cr, Fe, Al) O_4 ss

Chrome-alumina, 60-65%, (Cr, Fe, Al) O_4 ss + 35-40% (Mg, Cr) O_3

Zirconia-alumina, ~11% ZrO_2 , ~47% Al_2O_3 , ~11% SiO_2

Oxide ceramics, sintered:

Alumina, 85-98% Al_2O_3

Silicon oxy-nitride $\text{Si}_2\text{N}_2\text{O}$

Non-oxide ceramics:

Silicon carbide, SiC-sintered α , hot-pressed, sintered $\alpha+\beta$

Silicon carbide bonded with silicon nitride

Silicon nitride, Si_3N_4 -reaction-sintered, hot-pressed, sintered.

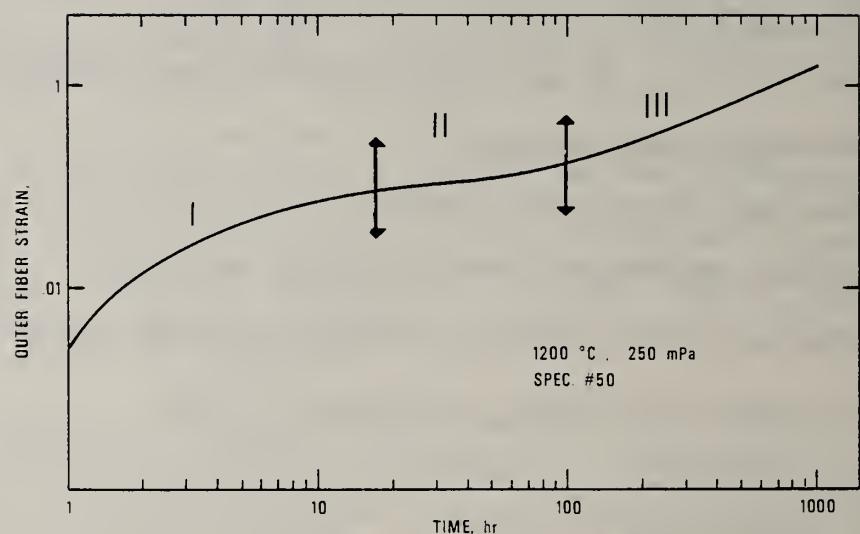


Figure 1. Plot of the outer fiber strain calculated from the deflection measured during 1000 hours at 1200 °C with a load of 250 MPa for hot-pressed silicon nitride (NC 132).